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NEWS 5 DEC 14 2006 MeSH terms loaded for MEDLINE file segment of TOXCENTER

NEWS 6 DEC 14 CA/CAplus to be enhanced with updated IPC codes

NEWS 7 DEC 21 IPC search and display fields enhanced in CA/CAplus with the IPC reform

NEWS 8 DEC 23 New IPC8 SEARCH, DISPLAY, and SELECT fields in USPATFULL/ USPAT2

NEWS 9 JAN 13 IPC 8 searching in IFIPAT, IFIUDB, and IFICDB

NEWS 10 JAN 13 New IPC 8 SEARCH, DISPLAY, and SELECT enhancements added to INPADOC

NEWS 11 JAN 17 Pre-1988 INPI data added to MARPAT

NEWS 12 JAN 17 IPC 8 in the WPI family of databases including WPIFV

NEWS EXPRESS JANUARY 03 CURRENT VERSION FOR WINDOWS IS V8.01,
CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),
AND CURRENT DISCOVER FILE IS DATED 19 DECEMBER 2005.
V8.0 USERS CAN OBTAIN THE UPGRADE TO V8.01 AT
http://download.cas.org/express/v8.0-Discover/

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=> s hexamethylenediamine or aminocapronitrile

9452 HEXAMETHYLENEDIAMINE

63 HEXAMETHYLENEDIAMINES

9486 HEXAMETHYLENEDIAMINE

(HEXAMETHYLENEDIAMINE OR HEXAMETHYLENEDIAMINES)

298 AMINOCAPRONITRILE

3 AMINOCAPRONITRILES

298 AMINOCAPRONITRILE

(AMINOCAPRONITRILE OR AMINOCAPRONITRILES)

9678 HEXAMETHYLENEDIAMINE OR AMINOCAPRONITRILE

=> s l1 and (process or prepar? or make or made or synthesi?)

2191689 PROCESS

L1

1474198 PROCESSES

3266134 PROCESS

(PROCESS OR PROCESSES)

1609165 PREPAR?

120098 PREP

2137 PREPS

122030 PREP

(PREP OR PREPS)

1979199 PREPD

21 PREPDS

1979214 PREPD

(PREPD OR PREPDS)

115677 PREPG

12 PREPGS

115688 PREPG

(PREPG OR PREPGS)

2667572 PREPN

202660 PREPNS

2820629 PREPN

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10/663,479
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(PREPN OR PREPNS) 4663249 PREPAR? (PREPAR? OR PREP OR PREPD OR PREPG OR PREPN) 222095 MAKE 173552 MAKES 383896 MAKE (MAKE OR MAKES) 1183939 MADE 24 MADES 1183960 MADE (MADE OR MADES) 1470529 SYNTHESI? 6682 L1 AND (PROCESS OR PREPAR? OR MAKE OR MADE OR SYNTHESI?) L2 => s 12 and adiponitrile 2055 ADIPONITRILE 63 ADIPONITRILES 2067 ADIPONITRILE (ADIPONITRILE OR ADIPONITRILES) L3 289 L2 AND ADIPONITRILE => s 13 and tetrahydroazepine 71 TETRAHYDROAZEPINE 16 TETRAHYDROAZEPINES 83 TETRAHYDROAZEPINE (TETRAHYDROAZEPINE OR TETRAHYDROAZEPINES) L46 L3 AND TETRAHYDROAZEPINE => s 13 and hexamethyleneimine 401 HEXAMETHYLENEIMINE 11 HEXAMETHYLENEIMINES 409 HEXAMETHYLENEIMINE (HEXAMETHYLENEIMINE OR HEXAMETHYLENEIMINES) 4 L3 AND HEXAMETHYLENEIMINE L5 => s 13 and raney nickel or raney cobalt 28002 RANEY 1 RANEYS 28002 RANEY (RANEY OR RANEYS) 594636 NICKEL 197 NICKELS 594663 NICKEL (NICKEL OR NICKELS) 4901 RANEY NICKEL (RANEY (W) NICKEL) 28002 RANEY 1 RANEYS 28002 RANEY (RANEY OR RANEYS) 362285 COBALT 95 COBALTS 362288 COBALT (COBALT OR COBALTS) 203 RANEY COBALT (RANEY (W) COBALT) L6 233 L3 AND RANEY NICKEL OR RANEY COBALT => s 13 and (raney nickel or raney cobalt)

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28002 RANEY
             1 RANEYS
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                 (RANEY OR RANEYS)
        594636 NICKEL
           197 NICKELS
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                 (NICKEL OR NICKELS)
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         28002 RANEY
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            95 COBALTS
        362288 COBALT
                 (COBALT OR COBALTS)
           203 RANEY COBALT
                 (RANEY (W) COBALT)
L7
            35 L3 AND (RANEY NICKEL OR RANEY COBALT)
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     (FILE 'HOME' ENTERED AT 17:34:10 ON 19 JAN 2006)
     FILE 'CAPLUS' ENTERED AT 17:34:22 ON 19 JAN 2006
           9678 S HEXAMETHYLENEDIAMINE OR AMINOCAPRONITRILE
L1
L2
           6682 S L1 AND (PROCESS OR PREPAR? OR MAKE OR MADE OR SYNTHESI?)
L3
            289 S L2 AND ADIPONITRILE
              6 S L3 AND TETRAHYDROAZEPINE
L4
              4 S L3 AND HEXAMETHYLENEIMINE
L5
L6
            233 S L3 AND RANEY NICKEL OR RANEY COBALT
L7
             35 S L3 AND (RANEY NICKEL OR RANEY COBALT)
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PROCESSING COMPLETED FOR L4
PROCESSING COMPLETED FOR L5
PROCESSING COMPLETED FOR L7
             44 DUP REM L4 L5 L7 (1 DUPLICATE REMOVED)
=> d 18 ibib hitstr abs 1-44
     ANSWER 1 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN DUPLICATE 1
ACCESSION NUMBER:
                         2005:238743 CAPLUS
DOCUMENT NUMBER:
                         142:298456
TITLE:
                         Process for producing
                         hexamethylenediamine and
                         aminocapronitrile from adiponitrile,
                         wherein the hexamethylenediamine contains
                         less than 100 ppm tetrahydroazepine
INVENTOR(S):
                         Allgeier, Alan Martin; Ostermaier, John J.
PATENT ASSIGNEE(S):
                         USA
SOURCE:
                         U.S. Pat. Appl. Publ., 4 pp.
                         CODEN: USXXCO
DOCUMENT TYPE:
                         Patent
LANGUAGE:
                         English
FAMILY ACC. NUM. COUNT:
PATENT INFORMATION:
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AB

L8

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PATENT NO.
                         KIND
                                  DATE
                                             APPLICATION NO.
                                                                       DATE
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                                              -----
                                              US 2003-663479
     US 2005059822
                           A1
                                  20050317
                                                                       20030915
                                            WO 2004-US30257
     WO 2005028418
                          A1
                                  20050331
                                                                       20040915
             AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI,
              NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY,
              TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
         RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM,
             AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK,
              EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE,
              SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE,
             SN, TD, TG
PRIORITY APPLN. INFO.:
                                              US 2003-663479
                                                                    A 20030915
     Process for making both ACN and HMD from partial hydrogenation
     of ADN by using a combination of distns. resulting in the formation of a
     mixture of HMD and THA that can be hydrogenated to produce a mixture of HMD
     and HMI that can be separated easily by simple distillation
     ANSWER 2 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER:
                          2004:1127152 CAPLUS
DOCUMENT NUMBER:
                          142:74968
TITLE:
                          Hydrolytic and distillation method for making
                          ε-caprolactam from impure 6-
                          aminocapronitrile in which
                          tetrahydroazepine is not removed until after
                          the \varepsilon-caprolactam is produced
INVENTOR(S):
                          Kirby, Gregory S.; Ostermaier, John J.
                          Invista North America S.A.R.L., USA
PATENT ASSIGNEE(S):
SOURCE:
                          U.S. Pat. Appl. Publ., 6 pp.
                          CODEN: USXXCO
DOCUMENT TYPE:
                          Patent
LANGUAGE:
                          English
FAMILY ACC. NUM. COUNT:
PATENT INFORMATION:
     PATENT NO.
                          KIND
                                  DATE
                                                                     DATE
                                            APPLICATION NO.
                                  -----
     US 2004260087
                                  20041223
                                            US 2003-464104
                          A1
                                                                       20030617
                          B2
     US 6858728
                                  20050222
     WO 2005000808
                          A1
                                  20050106
                                              WO 2004-US19442
                                                                       20040617
             AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH,
             CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD,
             GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC,
             LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI,
             NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY,
             TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
         RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM,
             AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK,
             EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE,
             SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE,
             SN, TD, TG
PRIORITY APPLN. INFO.:
                                              US 2003-464104
                                                                 A 20030617
     A method for making caprolactam from an impure 6-aminocapronitrile
     (ACN), obtained by the partial hydrogenation of adiponitrile,
```

which comprises 6-aminocapronitrile and both ACN and ≥500

ppm tetrahydroazepine and its derivs. (THA), comprises: (1) contacting the impure ACN comprising both ACN and THA with water at elevated temperature in the presence of a dehydration catalyst (e.g., alumina), both the impure ACN and the water being in the vapor phase, to produce a vapor-phase reaction product that comprises ε-caprolactam, ammonia, water, ACN, and THA; (2) separating the ammonia and a major portion of the water from the vapor-phase reaction product to produce a solution comprising &-caprolactam and a minor portion of the water, and then separating the water from the solution to produce a melt comprising E-caprolactam, ACN and THA; (3) introducing the melt into a low-boiler-removal distillation column and removing a major portion of both the THA and ACN as a distillate, and removing ε-caprolactam, high boilers and at most a minor portion of both the THA and ACN as a bottoms; and (4) introducing the bottoms into a high-boiler-removal distillation column and removing ϵ -caprolactam and at most a minor portion of the high boilers as a distillate product and removing a major portion of the high boilers as a bottoms. Process flow diagrams are presented. REFERENCE COUNT: THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS 5

L8ANSWER 3 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

2004:779941 CAPLUS

DOCUMENT NUMBER:

141:279773

TITLE:

Distillation method for separating

hexamethylenediamine from a mixture comprising

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

hexamethylenediamine, 6aminocapronitrile and tetrahydroazepine

INVENTOR(S):

Ostermaier, John J.

PATENT ASSIGNEE(S):

Invista North America S.A.R.L., USA

U.S. Pat. Appl. Publ., 5 pp.

CODEN: USXXCO

DOCUMENT TYPE:

Patent

LANGUAGE:

SOURCE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PA	TENT	NO.					DATE			APPL	I CAT	ION	NO.		D	ATE	
															-		
US	2004	1826	90		A1		2004	0923	1	US 2	003-3	3839	47		2	00303	307
US	6887	352			B2		2005	0503									
WO	2004	0809	32		A2		2004	0923	1	WO 2	004-1	JS64	63		2	00403	303
WO	2004	0809	32		A3		2005	0324		_							
	W:	ΑE,	AG,	AL,	AM,	AT,	AU,	AZ,	BA,	BB,	BG,	BR,	BW,	BY,	BZ,	CA,	CH,
							DE,										
							ID,										
							LV,										
							PL,										
							TZ,										
	RW:						MW,		-		•	•	•		•	•	
		BY,	KG,	ΚZ,	MD,	RU,	ТJ,	TM,	ΑT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,
		ES,	FI,	FR,	GB,	GR,	HU,	ΙE,	IT,	LU,	MC,	NL,	PL,	PT,	RO,	SE,	SI,
		SK,	TR,	BF,	ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GO,	GW,	ML,	MR.	NE.	SN,
		TD,		•	•	•	•	•	•	- •		- ~ '	- ,		,	,	,
EP	1603	651			A2		2005	1214		EP 2	004-	7168	81		2	0040	303
	R:	AT,	BE,	CH,	DE,	DK,	ES,	FR,	GB,	GR,	IT.	LI,	LU,	NL,	SE.	MC.	PT.
							RO,										
PRIORIT	Y APP				,	,	,									00303	
2.0101011				• •												00403	
									1	WO Z	004-1	1004	0.5	,	v 2	1040.	303

A method for recovering hexamethylene diamine (HMD) from a mixture AB comprising HMD, 6-aminocapronitrile (ACN) tetrahydroazepine (THA), and ADN comprises: (a) introducing the mixture into a distillation column capable of separating as a group the HMD, ACN and at

least a portion of the THA from the ADN, while minimizing the isomerization of the ADN into CPI; and (b) introducing the HMD, ACN and at least a portion of the THA into a distillation column capable of separating

from the ACN in such a way that the THA separates along with the ACN. 'REFERENCE COUNT: THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS 8 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

ANSWER 4 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN 1.8

ACCESSION NUMBER: 2003:585478 CAPLUS

DOCUMENT NUMBER: 139:137926

TITLE: Recovery of adiponitrile from a waste

> mixture of adiponitrile, aminocapronitrile and hexamethylenediamine

INVENTOR(S): Ostermaier, John; Scott, Leon; Hastings, James

PATENT ASSIGNEE(S): E. I. Du Pont de Nemours & Co., USA

SOURCE: U.S., 6 pp. CODEN: USXXAM

DOCUMENT TYPE: Patent English

LANGUAGE:

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

	PA'	rent :	NO.			KIN	D	DATE				I CAT				D	ATE	
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	WO	2004	0074	34		A1		2004	0122	1	WO 2	003-1	JS22	127		2	0030	715
		W:	AE,	AG,	AL,	AM,	AT,	AU,	AZ,	BA,	BB,	BG,	BR,	BY,	BZ,	CA,	CH,	CN,
			CO,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	ES,	FI,	GB,	GD,	GE,	GH,
			GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	KE,	KG,	KP,	KR,	KZ,	LC,	LK,	LR,
			LS,	LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NI,	NO,	NZ,	OM,
			PG,	PH,	PL,	PT,	RO,	RU,	SC,	SD,	SE,	SG,	SK,	SL,	SY,	TJ,	TM,	TN,
			TR,	TT,	TZ,	UA,	UG,	UZ,	VC,	VN,	YU,	ZA,	ZM,	ZW				
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			KG,	ΚZ,	MD,	RU,	TJ,	TM,	AT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,	ES,
			FI,	FR,	GB,	GR,	HU,	ΙE,	IT,	LU,	MC,	NL,	PT,	RO,	SE,	SI,	SK,	TR,
			BF,	ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	MR,	NE,	SN,	TD,	TG
	BR	2003	0128	97		Α		2005	0614]	BR 2	003-	1289	7		2	0030	715
	EP	1539	680			A1		2005	0615		EP 2	003-	7646	99		2	0030	715
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			ΙE,	SI,	LT,	LV,	FI,	RO,	MK,	CY,	AL,	TR,	BG,	CZ,	EE,	HU,	SK	
	JP	2005	5331	17		T2		2005	1104	,	JP 2	004-	5218	67		2	0030	715
PRIO	RIT	APP	LN.	INFO	. :					1	US 2	002-	1978	82	i	A 2	0020	717
										1	WO 2	003-1	JS22	127	1	W 2	0030	715
NΠ	n		for	+ha	~~~	~··~		~ ~	f									

AΒ Process for the recovery of a purified adiponitrile (ADN) from a mixture of adiponitrile, aminocapronitrile

and hexamethylenediamine, utilizing two sequential distns.: (1)

a first distillation in which the mixture is distilled in a distillation column at a head

pressure that causes at least 7% of the ADN to go into the distillate, along with bishexamethylenetriamine (BHMT) and 2-

cyanocyclopentylideneimine (CPI), and (2) a second distillation in which the distillate from the first distillation is distilled in a second distillation column at a

head pressure sufficient to cause min.-temperature azeotropy between ADN and BHMT, thereby allowing the majority of the BHMT and CPI to be removed from the second distillation as distillate, and ADN, substantially free of both BHMT and CPI, to be removed as bottoms.

REFERENCE COUNT: THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

ANSWER 5 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN L8

ACCESSION NUMBER: 2003:5923 CAPLUS

DOCUMENT NUMBER: 138:75102

TITLE: Method and catalysts for the hemihydrogenation of

dinitriles into aminonitriles

INVENTOR(S): Leconte, Philippe; Lopez, Joseph PATENT ASSIGNEE(S): Rhodia Polyamide Intermediates, Fr.

SOURCE: PCT Int. Appl., 11 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: French

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION: DATENT NO

PA	TENT	NO.			KIN	D	DATE				ICAT				D.	ATE	
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		GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	KE,	KG,	KP,	KR,	KZ,	LC,	LK.	LR,
		-	•				MD,			•		•	•	•	•	•	•
					-	-	SE,	-	-	-				•	•	•	
							YU,										
		ТJ,		•	•	•		•	•	•	,	•	•		•	•	•
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							CM,							-		-	
FR	2826																
	2826																
CA	2449	121			AA		2003	0103		CA 2	002-	2449	121		2	0020	613
EF	1397	346			A2		2004	0317		EP 2	002-	7808	41		2	0020	613
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	1518				Α		2004	0804		CN 2	002-	8125	00		2	0020	613
BR	2002	0110	14		Α		2004	0810		BR 2	002-	1101	4		2	0020	613
JF	2004	5307	19		T2		2004	1007	,	JP 2	003-	5070	58		2	0020	613
ŔŬ	2260	587			C1		2005	0920	:	RU 2	004-	1016	04		2	0020	613
US	2004						2004									0040	527
PRIORIT	Y APP	LN.	INFO	. :						FR 2	001-	8245		i	A 2	0010	622
									1	WO 2	002-1	FR202	23	1	<i>N</i> 2	0020	613

MARPAT 138:75102

The hemihydrogenation of dinitriles (e.g., adiponitrile) into the corresponding aminonitriles (e.g., aminocapronitrile) is described using water and a hydrogenation catalyst system (e.g., Raney nickel, KOH, and Et4NF) containing selecting agents.

ANSWER 6 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2002:730590 CAPLUS

DOCUMENT NUMBER: 137:249499

TITLE: Hydrogenation process and catalyst systems

for the manufacture of aminonitriles from dinitriles

INVENTOR(S): Ionkin, Alex Sergey

PATENT ASSIGNEE(S): E. I. Du Pont de Nemours & Co., USA

SOURCE: U.S., 7 pp.
CODEN: USXXAM

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PA'	TENT :	NO.			KIN	D	DATE			APPL	ICAT	ION 1	NO.		D	ATE	
						-									-		
US	6455	723			B1		2002	0924	1	US 2	001-	6826	56		2	0011	002
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		ΡĹ,	PT,	RO,	RU,	SD,	SE,	SG,	SI,	SK,	SL,	TJ,	TM,	TN,	TR,	TT,	TZ,
		UA,	UG,	US,	UZ,	VC,	VN,	YU,	ZA,	ZM,	ZW						
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		FI,	FR,	GB,	GR,	ΙE,	IT,	LU,	MC,	NL,	PT,	SE,	SK,	TR,	BF,	ВJ,	CF,
		CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	MR,	NE,	SN,	TD,	TG			
EP	1434	758			A1		2004	0707		EP 2	002-	7786	07		2	0021	002
	R:	AT,	BE,	CH,	DE,	DK,	ES,	FR,	GB,	GR,	ΙT,	LI,	LU,	NL,	SE,	MC,	PT,
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CN	1556	789			Α		2004	1222		CN 2	002-	8183	34		2	0021	002
PRIORIT	Y APP	LN.	INFO	. :					1	US 2	001-	6826	56	1	A 2	0011	002
									1	WO 2	002-	US33:	255	1	W 2	0021	002
		\															

OTHER SOURCE(S): MARPAT 137:249499

AB A process for the high-yield partial hydrogenation of a dinitrile (e.g., adiponitrile) into an aminonitrile (e.g., 6-aminocapronitrile) is described comprising: contacting the dinitrile with a hydrogen-containing fluid in the presence of (A) a solvent comprising liquid ammonia, an alc. or both; (B) a hydrogenation catalyst (e.g., Raney cobalt); and (C) an effective amount of an additive comprising a compound selected from a divalent sulfur and a divalent selenium compound (e.g., selenophene).

REFERENCE COUNT: 19 THERE ARE 19 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 7 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2002:312051 CAPLUS

DOCUMENT NUMBER: 136:325981

TITLE: Catalyst system and process for the

hydrogenation of dinitriles into diamines and

aminonitriles

INVENTOR(S): Allgeier, Alan M.; Koch, Theodore A.; Sengupta, Sourav

Κ.

PATENT ASSIGNEE(S): E. I. Du Pont de Nemours & Co., USA

SOURCE: U.S., 6 pp. CODEN: USXXAM

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE

FR 2806081

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US 2001-871102
     US 6376714
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                                 20020423
                                                                    20010531
     TW 593235
                                 20040621
                                             TW 2002-91110365
                          В
                                                                    20020517
     CA 2444442
                          AA
                                 20021205
                                             CA 2002-2444442
                                                                    20020524
     WO 2002096862
                          A2
                                 20021205
                                             WO 2002-US16374
                                                                    20020524
     WO 2002096862
                          A3
                                 20030731
            AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
             CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
             GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,
             LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH,
             PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ,
             UA, UG, UZ, VN, YU, ZA, ZM, ZW
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             GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA,
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                                 20040303
                                            EP 2002-739372
     EP 1392646
                          A2
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     BR 2002010082
                                             BR 2002-10082
                          Α
                                 20040817
                                                                    20020524
     CN 1531523
                                 20040922
                                             CN 2002-810915
                          Α
                                                                    20020524
     JP 2004534778
                          T2
                                 20041118
                                             JP 2003-500042
                                                                    20020524
PRIORITY APPLN. INFO.:
                                             US 2001-871102
                                                                 A 20010531
                                                                 W 20020524
                                             WO 2002-US16374
     A process for converting dinitriles into diamines and/or
AR
     aminonitriles consists of forming a reaction mixture that comprises: (1) a
     dinitrile; (2) hydrogen; (3) a catalyst comprising a Group VIII element;
     and (4) one or more modifiers selected from quaternary ammonium
     hydroxides, quaternary ammonium cyanides, quaternary ammonium fluorides,
     quaternary phosphonium hydroxides, and quaternary ammonium thiocyanides.
     The reaction mixture contains less than a 1:1 molar ratio of solvent and the
     process is carried out at a pressure and temperature sufficient to
     convert at least a portion of the dinitrile (e.g., 1,6-hexanedinitrile)
     into a diamine (e.g., 1,6-diaminohexane) and, optionally, an aminonitrile
     (e.g., 6-aminocapronitrile).
REFERENCE COUNT:
                         3
                               THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS
                               RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT
     ANSWER 8 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER:
                         2001:676740 CAPLUS
DOCUMENT NUMBER:
                         135:227379
TITLE:
                         Method and catalyst for hydrogenating nitriles into
                         amines or aminonitriles
INVENTOR(S):
                         Boschat, Vincent; Leconte, Philippe
PATENT ASSIGNEE(S):
                         Rhodia Polyamide Intermediates, Fr.
SOURCE:
                         PCT Int. Appl., 21 pp.
                         CODEN: PIXXD2
DOCUMENT TYPE:
                         Patent
LANGUAGE:
                         French
FAMILY ACC. NUM. COUNT:
PATENT INFORMATION:
     PATENT NO.
                         KIND
                                DATE
                                            APPLICATION NO.
                                                                    DATE
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                                            -----
     WO 2001066511
                          A1
                                20010913
                                          WO 2001-FR687
                                                                    20010307
        W: AU, BR, BY, CA, CN, CZ, ID, IL, IN, JP, KR, MX, PL, RO, RU, SG, SK, TR, UA, US, VN, ZA
         RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
             PT, SE, TR
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FR 2000-2997

20000308

20010914

A1

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FR 2806081
                           B1
                                 20030314
     CA 2403210
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                                 20010913
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                                                                      20010307
     EP 1265845
                                             EP 2001-913956
                          A1
                                 20021218
                                                                      20010307
         R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR
                                 20030603
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                          T2
    JP 2003525924
                                 20030902
                                             JP 2001-565331
                                                                      20010307
    RU 2242460
                          C2
                                 20041220 RU 2002-126613
                                                                      20010307
                          A1
                                             US 2003-220821
    US 2003144552
                                 20030731
                                                                      20030110
                          B2
    US 6790994
                                 20040914
PRIORITY APPLN. INFO.:
                                             FR 2000-2997
                                                                  A 20000308
                                             WO 2001-FR687
                                                                  W 20010307
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A method for hydrogenating nitriles into amines, as well as the total or partial hydrogenation of dinitriles into diamines or aminonitrile compds., is described using hydrogen in the presence of a hydrogenation catalyst (e.g., Raney nickel containing Co) and a strong mineral

base (e.g., KOH) preferably derived from an alkaline or alkaline-earth metal.

The

catalyst used is subjected to conditioning by mixing the hydrogenation catalyst, a specific amount of strong mineral base, and a solvent in which the strong mineral base is hardly soluble The solvent is an amine compound such as hexamethylenediamine in the case of hydrogenation of

adiponitrile into HMD and/or aminocapronitrile.

REFERENCE COUNT: 8 THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

ANSWER 9 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN L8

ACCESSION NUMBER: 2001:439662 CAPLUS

DOCUMENT NUMBER: 135:210668

TITLE: Reactivity and surface analysis studies on the

deactivation of Raney Ni during adiponitrile

hydrogenation

AUTHOR (S): Allgeier, Alan M.; Duch, Michael W.

CORPORATE SOURCE: E.I. duPont de Nemours Co., Wilmington, DE, 19880, USA

Chemical Industries (Dekker) (2001), 82 (Catalysis of SOURCE:

Organic Reactions), 229-239

CODEN: CHEIDI; ISSN: 0737-8025

Marcel Dekker, Inc. PUBLISHER:

DOCUMENT TYPE: Journal LANGUAGE: English

The heterogeneous catalyst, Raney Ni, deactivates during the hydrogenation

of adiponitrile. The present study shows that the deactivation

process is general to α , ω -dinitriles of varying

length and also occurs for 6-aminocapronitrile but does not

occur with mononitriles such as butyronitrile. In contrast to a previously reported mechanism for Ni catalyst deactivation in acetonitrile hydrogenation, these reactivity trends implicate deposition of oligomeric secondary amines and thus blocking of active sites as the mechanism of deactivation. Electron spectroscopy for chemical anal. (ESCA) reveals an

increase in C and N on deactivated samples compared to nondeactivated samples and supports the conclusions drawn from reactivity studies.

REFERENCE COUNT: 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

ANSWER 10 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

2000:441758 CAPLUS ACCESSION NUMBER:

DOCUMENT NUMBER: 133:75638

TITLE: Method for preparing amines by hydrogenating

nitriles in reactor

INVENTOR(S): Goodwin, Ralph T., III; Ward, Gregory J.

PATENT ASSIGNEE(S): Solutia Inc., USA SOURCE: PCT Int. Appl., 16 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

	PA	rent 1	NO.			KIN		DATE				LICAT				D.	ATE	
	WO	2000	0374	24								1999-1				1	9991	210
		W:	ΑE,	AL,	AM,	AT,	AU,	AZ,	BA,	BB,	BG	, BR,	BY,	CA,	CH,	CN,	CR,	CU,
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			IN,	IS,	JP,	KE,	KG,	ΚP,	KR,	ΚZ,	LC	, LK,	LR,	LS,	LT,	LU,	LV,	MA,
			MD,	MG,	MK,	MN,	MW,	MX,	NO,	NZ,	PL	, PT,	RO,	RU,	SD,	SE,	SG,	SI,
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			BY,	KG,	KZ,	MD,	RU,	TJ,	TM									
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			CG,	CI,	CM,	GA,	GN,	GW,	ML,	MR,	NE	, SN,	TD,	TG				
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	EP	1169	296			A1		2002	0109	1	EP :	1999-	9661	30		1	9991:	210
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			ΙE,	SI,	LT,	LV,	FI,	RO										
	JP	2002	5333	19		Т2		2002	1008		JP :	2000-	5894	96		1	9991	210
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	RU	2233	266			C2		2004	0727	1	RU :	2001-	1203	44		1	9991	210
	US	6281	388			В1		2001	0828	1	US :	1999-	4600!	51		1	9991	213
PRIO	RIT	Y APP	LN.	INFO	.:					1	US :	1998-	1133	29P		P 1	9981	222
										Ī	WO :	1999-1	US29:	394	1	W 1	9991	210
						_			-									

AB The method comprises feeding hydrogen and a nitrile (e.g., adiponitrile) into a reactor having a reaction medium containing a catalyst (e.g., Raney nickel), water and an inorg. base (e.g., alkali metal hydroxide); mixing with the reaction medium to give a uniform bulk concentration of the nitrile in ≥1 direction across the reactor to minimize the reactor volume; and hydrogenating the nitrile to form the amine (e.g., hexamethylenediamine). The reactor comprises a stirred tank reactor, a gas lift reactor, a tubular reactor or a bubble column reactor.

REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 11 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2000:335376 CAPLUS

DOCUMENT NUMBER: 132:349278

TITLE: Partial hydrogenation of dinitriles to amino nitriles

INVENTOR(S): Boschat, Vincent; Brunelle, Jean-Pierre PATENT ASSIGNEE(S): Rhodia Fiber and Resin Intermediates, Fr.

SOURCE: PCT Int. Appl., 17 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE

EP 1137484

EP 1137484

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20000518 WO 1999-FR2677
    WO 2000027806
                       A1
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            US, VN
        RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
            PT, SE
                                        FR 1998-14100
    FR 2785608
                        A1
                              20000512
                                                                19981105
                       B1
    FR 2785608
                              20001229
                       AA
                              20000518
                                         CA 1999-2349928
    CA 2349928
                                                                19991103
    BR 9915085
                              20010717 BR 1999-15085
                                                                19991103
                       Α
                                       EP 1999-954042
    EP 1127047
                              20010829
                                                                19991103
                       A1
    EP 1127047
                       B1
                              20030910
        R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
            IE, FI, RO
    JP 2002539074
                       T2
                              20021119
                                          JP 2000-580986
                                                                19991103
    AT 249425
                       Ε
                              20030915 AT 1999-954042
                                                                19991103
                              20031227 RU 2001-115092
    RU 2220133
                       C2
                                                                19991103
                       Т3
                                       ES 1999-954042
    ES 2201795
                              20040316
                                                                19991103
                       В1
    US 6521779
                                       US 2001-831148
                              20030218
                                                                20010925
                                          TW 1999-88119218
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                              20011121
                                                                20011009
                                          FR 1998-14100 A 19981105
WO 1999-FR2677 W 19991103
                                                             A 19981105
PRIORITY APPLN. INFO.:
    A dinitrile is hydrogenated to the corresponding amino nitrile in a liquid
    medium in the presence of a Raney nickel or cobalt
    catalyst containing Cu and/or Ag and/or Au and in the presence of an alkali or
    alkaline earth metal hydroxide. Thus, hydrogenation of a mixture of
    adiponitrile 24.0, hexamethylenediamine 24.0, H2O 5.3,
    KOH 0.064, and Raney Ni (1.7% Cu) 1.35 g at 50° under 2.5 MPa H for
    321 min (to optimum yield) resulted in 82.3% conversion of
    adiponitrile: 60.3% to 6-aminocapronitrile and 20.9% to
    hexamethylenediamine.
REFERENCE COUNT:
                              THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS
                              RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT
    ANSWER 12 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER:
                     2000:335307 CAPLUS
DOCUMENT NUMBER:
                       132:336095
TITLE:
                       Raney cobalt catalysts and
                       process for hydrogenating organic compounds
                       using them
INVENTOR(S):
                       Harper, Mark Jay
                       E. I. Du Pont de Nemours & Co., USA
PATENT ASSIGNEE(S):
SOURCE:
                        PCT Int. Appl., 26 pp.
                        CODEN: PIXXD2
                        Patent
DOCUMENT TYPE:
LANGUAGE:
                        English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
    PATENT NO.
                      KIND DATE
                                        APPLICATION NO. DATE
                       _ _ _ _
    WO 2000027526
                       A1 20000518 WO 1999-US25952 19991104
        W: BR, CA, CN, ID, JP, KR, MX, SG
        RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
            PT, SE
    US 6156694
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A1
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    CA 2345523
                              20000518
                                                                19991104
                                          EP 1999-956905
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19991104

20011004

20030924

В1

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI

BR 9915248 A 20011030 BR 1999-15248 19991104 JP 2002529227 T2 20020910 JP 2000-580746 19991104 PRIORITY APPLN. INFO.: US 1998-186839 A 19981105 WO 1999-US25952 W 19991104

AB The catalysts comprise iron, cobalt, and a third metal selected from the group consisting of nickel, rhodium, ruthenium, palladium, platinum, osmium, iridium and mixts. of any of these metals. In the examples nickel is the third metal and the catalyst is used for hydrogenation of adiponitrile to 6-aminocapronitrile and

hexamethylenediamine.

REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 13 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1999:576901 CAPLUS

DOCUMENT NUMBER: 131:185369

TITLE: Method and catalysts for hydrogenating aliphatic

 α, ω -dinitriles into diamines or

aminonitriles

INVENTOR(S): Voit, Guido; Ohlbach, Frank; Luyken, Hermann; Merger,

Martin; Rehfinger, Alwin; Fischer, Rolf Hartmuth;

Bassler, Peter; Ansmann, Andreas

PATENT ASSIGNEE(S): Basf A.-G., Germany SOURCE: PCT Int. Appl., 16 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

	TENT						DATE		1	APP	LICA	TION	NO.		D	ATE	
WO	9944 9944	982					1999 1999		1	WO	1999	-EP11	49		1	9990	223
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	RW:	•	BE,	•	•		•	•		FR	R, GB	, GR,	IE,	IT,	LU,	MC,	NL,
DE	1980	9686			A1		1999	0909]	DE	1998	-1980	9686		1	9980	306
CA	2322	530			AA		1999	0910	4	CA	1999	-2322	530		1	9990	223
AU	9934	086			A1		1999	0920		ΑU	1999	-3408	6		1	9990	223
₿R	9908	504			Α		2000	1205	1	BR	1999	-8504			1	9990	223
EP	1058	677			A2		2000	1213		ΕP	1999	-9155	33		1	9990	223
EP	1058	677			B1		2003	0108									
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JP	2002	5053	15		T2		2002	0219		JΡ	2000	-5345	27		1	9990	223
ES	2190	648			Т3		2003	0801		ES	1999	-9155	33		1	9990	223
TW	5846	20			В		2004	0421	•	TW	1999	-8810	3427		1	9990	305
US	6265	602			B1		2001	0724	1	US	2000	-6228	00		2	0000	823
PRIORIT	Y APP	LN.	INFO	. :								-1980 -EP11				9980 9990	

AB Aliphatic α, ω -dinitriles (e.g., adipodinitrile) in the presence of a heterogeneous fixed-bed catalyst are hydrogenated into their corresponding diamines (e.g., 1,6-hexanediamine) or aminonitrile products with reduced formation of unwanted cyclic byproducts (e.g., tetrahydroazepine). The method is characterized in that the

reaction mixture contains 2-30 mmol Na, K, Rb, Cs, Mg, Ca, Sr, Ba, Mn, or their mixts. in the form of a basic salt, in relation to 10 mol of the aliphatic α, ω -dinitriles.

ANSWER 14 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

1999:753204 CAPLUS ACCESSION NUMBER:

DOCUMENT NUMBER: 132:3589

TITLE: Method for preparing amino nitriles and

diamines

Leconte, Philippe INVENTOR (S):

PATENT ASSIGNEE(S): Rhodia Fiber and Resin Intermediates, Fr.

SOURCE: PCT Int. Appl., 14 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PA	TENT NO.			KINI	D DATE	APPLICATION NO.	DATĖ
WC	9959962			A1		WO 1999-FR1127	
	RW: AT					JP, KR, PL, RO, RU, FI, FR, GB, GR, IE,	
FR	2778661			A1	19991119	FR 1998-6426	19980515
FF	2778661			B1	20000616		
CA	2332613			AA	19991125	CA 1999-2332613	19990511
BF	9910472			Α	20010109	BR 1999-10472	19990511
EF	1077932			A1	20010228	EP 1999-918054	19990511
EF	1077932			B1	20030716		
	R: BE,	, DE,	ES,	FR,	GB, IT, NL		
JF	20025154	178		T2	20020528	JP 2000-549581	19990511
RU	2210564			C2	20030820	RU 2000-131622	19990511
ES	2198910			Т3	20040201	ES 1999-918054	19990511
TW	1 487694			В	20020521	TW 1999-88107919	19990515
US	6384283			B1	20020507	US 2001-674551	20010126
PRIORIT	Y APPLN.	INFO	.:			FR 1998-6426 WO 1999-FR1127	

In preparation of an amino nitrile and a diamine by catalytic AB hydrogenation of a C3-12 aliphatic dinitrile, the final reaction mixture, from which the catalyst has been separated, is acidified with a mineral or organic acid before distilling the reaction products and the unreacted dinitrile. More specifically, the process concerns the preparation of

6-aminocapronitrile and hexamethylenediamine by

hydrogenating adiponitrile. This method suppresses the

formation of iminocyanocyclopentane as a byproduct.

REFERENCE COUNT: THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

ANSWER 15 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1999:354474 CAPLUS

DOCUMENT NUMBER: 130:353934

TITLE: Method and catalysts for the continuous hydrogenation

of dinitriles into aminonitriles

Boschat, Vincent; Leconte, Philippe; Rochette, Daniel; INVENTOR(S):

Sever, Lionel

Rhodia Fiber and Resin Intermediates, Fr. PATENT ASSIGNEE(S):

PCT Int. Appl., 23 pp. SOURCE:

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: French

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

	PATE	ON TV						DATE		F	APF	LIC	CAT	I NO	10.		D	ATE	
	WO 99							1999	0603	- V	10	199	 98-I	R24	- - 79		1	9981	119
	V	v : B	R,	BY,	CA,	CN,	CZ,	ID,	JP,	KR,	ΡI	, F	RO,	RU,	SG,	SK,	UA,	US,	VN
		RW: A																	
			T,		•	•				•		•		•	•	•	•	·	•
	FR 27	77109	1			A1		1999	0521	F	PR.	199	97-1	14809	9		1	9971	120
	FR 27	77109	1			В1		2000	0114										
	CA 23	31014	5			AA		1999	0603	(ĽΑ	199	98-2	2310	L45		1	9981	119
	EP 10	3255	8			A1		2000	0906	E	ΞP	199	98-9	95570)2		1	9981	119
	EP 10	3255	8			B1		2004	0225										
	F	R: B	ΒE,	DE,	ES,	FR,	GB,	IT,	NL										
	JP 20	0152	446	4		T2		2001	1204	Ċ	JΡ	200	00-5	5220	75		1	9981	119
	RU 21	18171	.6			C2		2002	0427	F	₹Ū	200	00-1	11558	35		1	9981	119
	CN 13	L1707				В		2003	0806	(CN	199	98-8	31274	12		1	9981	119
	TW 46	57884				В		2001	1211	7	ľW	199	98-8	37119	9263		1	9981	120
	BR 20	00000	257	3		Α		2002	0205	E	3R	200	00-2	2573			2	0000	602
	US 62	23248	8			B1		2001	0515	Ţ	JS	200	00-5	55488	37		2	0000	919
PRIO	RITY A	APPLN	i. I	NFO.	. :					F	PR.	199	97-1	14809	9	1	A 1	9971	120
										И	10	199	98 - B	R24	79	Į	W 1	9981	119

OTHER SOURCE(S): MARPAT 130:353934

Aliphatic dinitriles (e.g., adiponitrile) are continuously hydrogenated into their corresponding aminonitriles (e.g., aminocapronitrile) in the presence of a hydrogenation catalyst

(e.g., Raney nickel) nondissolved in the reaction

medium. The hydrogenation is carried out in a reactor capable of separating the hydrogenate and the catalyst in a zone where the gas-liquid transfer is limited or nonexistent, said separation and recycling of the catalyst being carried out in a time interval of ≤30 min. REFERENCE COUNT:

ANSWER 16 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1999:694948 CAPLUS

8

DOCUMENT NUMBER: 132:37222

TITLE: Selective hydrogenation of adiponitrile over

a Raney Ni-P amorphous catalyst

AUTHOR (S): Li, Hexing; Xu, Yeping; Deng, Jing-Fa

CORPORATE SOURCE: Department of Chemistry, Fudan University, Shanghai.

Peop. Rep. China

SOURCE: New Journal of Chemistry (1999), 23(11), 1059-1061

CODEN: NJCHE5; ISSN: 1144-0546

Royal Society of Chemistry PUBLISHER:

DOCUMENT TYPE: Journal LANGUAGE: English

Selective hydrogenation of adiponitrile to

hexamethylenediamine was carried out using Raney Ni-P amorphous

catalyst.

REFERENCE COUNT: 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

ANSWER 17 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1998:558759 CAPLUS

DOCUMENT NUMBER: 129:161948

TITLE: Distillative method for the separation of an imine

from a mixture containing an amine and an imine

INVENTOR(S): Luyken, Hermann; Bassler, Peter; Rehfinger, Alwin

PATENT ASSIGNEE(S): BASF A.-G., Germany SOURCE: Ger. Offen., 4 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent LANGUAGE: German

FAMILY ACC. NUM. COUNT: 2

PATENT INFORMATION:

PAT	CENT N	O.			KIN	D DATE	1	AP	PLICAT	ION I	NO.		D	ATE		
DE	19704	614			A1	 1998	0813	DE	1997-	1970	4614		1:	99702	207	
CA	22793	69			AA	1998	0813	CA	1998-	22793	369		1:	9980	130	
WO	98349	00			A1	1998	0813	WO	1998-	EP504	4		1:	9980	130	
	W :	AL,	AU,	BG,	BR,	BY, CA,	CN,	CZ, G	E, HU,	ID,	IL,	JP,	KR,	KZ,	LT,	
		LV,	MX,	NO,	NZ,	PL, RO,	RU,	SG, S	I, SK,	TR,	UA,	US,	AM,	ΑZ,	BY,	
		KG,	KZ,	MD,	RU,	TJ, TM										
	RW:	ΑT,	BE,	CH,	DE,	DK, ES,	FI,	FR, G	B, GR,	ΙE,	IT,	LU,	MC,	NL,	PT,	SE
AU	98609	81			A1	1998	0826	AU	1998-	60983	1		1:	9980	130	
EP	96484	7			A1	1999	1222	EP	1998-	9053	74		1	9980	L30	
EP	96484	7			B1	20.02	0102									
	R:	BE,	DE,	ES,	FR,	GB, IT,	NL									
JP	20015	1242	29		T2	2001	0821	JP	1998-	53372	25		1	9980	L30	
ES	21704	76			T3	2002	0801	ES	1998-	9053	74		1	9980	L30	
TW	48645	8			В	2002	0511	TW	1998-	8710	1636		1	99802	207	
US	62521	15			B1	2001	0626	US	1999-	34194	48		19	9990	721	
PRIORITY	APPL	N. 3	INFO	. :				DE	1997-	19704	4614	1	A 19	99702	207	
								WO	1998-	EP504	4	1	N 1	9980	130	

AB Imines (e.g., tetrahydroazepine) are removed either partially or totally from mixts. containing an amine (e.g., hexamethylenediamine) and an imine by the addition of an inert compound (e.g., adiponitrile) having a b.p. above that of the amine and then distilling the mixture so as to

produce a purified amine overhead product and a bottoms product containing the inert compound and the imine.

L8 ANSWER 18 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1998:118626 CAPLUS

DOCUMENT NUMBER: 128:141170

TITLE: Simultaneous preparation of caprolactam and

hexamethylenediamine from adiponitrile

INVENTOR(S): Bassler, Peter; Luyken, Hermann; Achhammer, Gunther;

Witzel, Tom; Fuchs, Eberhard; Fischer, Rolf; Schnurr,

Werner

PATENT ASSIGNEE(S): BASF A.-G., Germany

SOURCE: U.S., 11 pp., Cont.-in-part of U.S. Ser. No. 375,574,

abandoned.
CODEN: USXXAM

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 2

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5717090	Α	19980210	US 1995-565214	19951130
DE 19500222	Al	19960711	DE 1995-19500222	19950105

AU 9713696

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DE 1995-19500222 A 19950105
US 1995-375574 B2 19950118
PRIORITY APPLN. INFO.:
                                             US 1995-375574
                                                                  B2 19950118
     The title process is disclosed, in which (a)
     adiponitrile is partially hydrogenated to give a mixture containing
     essentially 6-aminocapronitrile, hexamethylenediamine,
     ammonia, adiponitrile and hexamethyleneimine; (b) the
     mixture obtained in (a) is subjected to a distillation to give ammonia as the
top
     product and a bottom product I, (c) the bottom product I containing
     essentially 6-aminocapronitrile, hexamethylenediamine,
     adiponitrile, hexamethyleneimine, inert compound A and
     ammonia, the ammonia content being lower than that of the mixture used in
     stage (b), is subjected to a second distillation to give a mixture comprising
the
     inert compound A and ammonia as the top product and a bottom product II, (d)
     the bottom product II is subjected, in a third column, to a distillation to
give
     the inert compound A as the top product and a bottom product III, (e) the
     bottom product III is subjected, in a fourth column, to a distillation to give
     top product KP1, containing essentially hexamethyleneimine and a
     bottom product IV, (f) the top product KP1 is subjected, in a fifth
     column, to a distillation to give a top product KP2, which contains essentially
     hexamethyleneimine, and (g) the bottom product IV containing
     essentially 6-aminocapronitrile and adiponitrile is
     subjected, in a sixth column, to a distillation to give 6-
     aminocapronitrile. The 6-aminocapronitrile thus
     obtained is then cyclized to give caprolactam.
REFERENCE COUNT:
                         1
                               THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS
                                RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT
     ANSWER 19 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER:
                       1997:500173 CAPLUS
DOCUMENT NUMBER:
                         127:109324
TITLE:
                         Manufacture of caprolactam and
                         hexamethylenediamine simultaneously from
                          adiponitrile
INVENTOR (S):
                         Achhammer, Guenther; Basler, Peter; Fischer, Rolf;
                         Fuchs, Eberhard; Luyken, Hermann; Schnurr, Werner;
                         Voit, Guido; Hilprecht, Lutz
PATENT ASSIGNEE(S):
                         BASF A.-G., Germany
SOURCE:
                         Ger. Offen., 12 pp.
                         CODEN: GWXXBX
DOCUMENT TYPE:
                         Patent
LANGUAGE:
                         German
FAMILY ACC. NUM. COUNT:
PATENT INFORMATION:
     PATENT NO. KIND DATE APPLICATION NO. DATE
                                 19970626 DE 1995-19548289 19951222
19970703 CA 1996-2237727 19961211
     DE 19548289 A1
CA 2237727 AA
CA 2237727 C
                  C
A1
     CA 2237727
                                 20040224
                              19970703 WO 1996-EP5521
     WO 9723454
         W: AU, BG, BR, BY, CA, CN, CZ, FI, GE, HU, IL, JP, KR, KZ, LV, MX, NO, NZ, PL, RO, RU, SG, SI, SK, TR, UA, US, AM, AZ, BY, KG, KZ,
             MD, RU, TJ, TM
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A1

RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE

19970717 AU 1997-13696 19961211

EP 876341 EP 876341	A1 B1			1996-943913		19961211
R: BE	, DE, ES, FR,	GB, IT, NL				
BR 9612107	A	19990223	BR	1996-12107		19961211
JP 2000502	660 T2	20000307	JP	1997-523263		19961211
ES 2149514	Т3	20001101	ES	1996-943913		19961211
US 6147208	A	20001114	US	1998-91130		19980616
CN 1375489	A	20021023	CN	2001-132635		20010905
PRIORITY APPLN.	INFO.:		DE	1995-19548289	Α	19951222
			WO	1996-EP5521	W	19961211

AB Adiponitrile (I) is partially hydrogenated, and the product containing 6-aminocapronitrile (II), hexamethylenediamine (III), NH3, I, and hexamethyleneimine is passed through 5 fractionation columns to sep. out III and II, of which II is cyclized in a further step to caprolactam.

L8 ANSWER 20 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1998:1407 CAPLUS

DOCUMENT NUMBER: 128:50297

TITLE: Method for filtering a three-phase catalytic reaction

mixture

INVENTOR(S): Perrona, Philippe; Sever, Lionel

PATENT ASSIGNEE(S): Rhone-Poulenc Fiber and Resin Intermediates, Fr.

SOURCE: PCT Int. Appl., 13 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

		APPLICATION NO.	
WO 9746306 W: BR, BY, CA,	A1 19971211 CN, CZ, JP, KR,	WO 1997-FR937 MX, PL, RO, RU, SG, SF FR, GB, GR, IE, IT, LU	19970529 K, UA, US, VN
FR 2749191	A1 19971205	FR 1996-7169	
FR 2749191			
EP 925106	A1 19990630	EP 1997-926054	19970529
EP 925106			
R: BE, DE, FR,	GB, IT, NL, RO		
CN 1223600	A 19990721	CN 1997-195789	19970529
CN 1112230	B 20030625		
BR 9709525	A 19990810	BR 1997-9525 JP 1998-500260	19970529
JP 2000500701	T2 20000125	JP 1998-500260	19970529
JP 3311359			
		RU 1999-100051	
		PL 1997-330310	
CZ 291317		CZ 1998-3961	
CA 2257346		CA 1997-2257346	19970529
CA 2257346			
SK 283673		SK 1998-1661	
MX 9810151	A 20000131	MX 1998-10151	19981202
		KR 1998-709935	
US 6478968	B1 20021112	US 1999-194907	19990317
PRIORITY APPLN. INFO.:		FR 1996-7169 WO 1996-FR7169	A 19960604
		WO 1996-FR7169	W 19960604
		WO 1997-FR937	W 19970529

AB A three-phase reaction mixture comprising a liquid phase containing nitriles, an

undissolved solid catalytic phase comprising Raney Ni and/or Co or supported metal catalysts and a gas phase (H2) is filtered tangentially using a single membrane filter for recycling the active catalyst while recovering the filtrate containing the reaction products. The hydrogenation reaction mixture containing nitriles, aminonitriles, and amines, H2, and Ni or Co or supported metal catalysts is filtered using a nanoporous or microporous membrane on a flat or tubular support, preferably ceramic, with active layer from α-alumina, zirconia, titania, or graphite fibers on a graphite, alumina, zirconia or titania support. In an example, a mixture containing adiponitrile 20.1%, aminocapronitrile 51.4%, hexamethylenediamine 9.1%, water 14.2%, and Raney nickel 5.2% under H2 at 2 bar and 55°C was pumped past a zirconia membrane on graphite support with average pore diameter 25-50 nm and 300 kD cutoff, and the catalyst-containing

retentate was returned to the reactor. The permeate was recovered at atmospheric

pressure.

L8 ANSWER 21 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1997:317793 CAPLUS

DOCUMENT NUMBER: 126:294889

TITLE: Metallic compounds useful as catalysts and their

preparation

INVENTOR(S): Cordier, Georges; Popa, Jean-Michel

PATENT ASSIGNEE(S): Rhone-Poulenc Fiber and Resin Intermediates, Fr.;

Cordier, Georges; Popa, Jean-Michel

SOURCE: PCT Int. Appl., 23 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PAT	CENT NO	•		KIN		DATE		AP	PLICAT	ION NO.		Ι	DATE		
WO	97 1 005 W: B			A1				WO	1996-	FR1406]	.99609	912	
								FR. G	B. GR.	IE, IT	LU.	MC.	NI.	PT.	SE
FR	273875									11064					-
FR	273875	7		B1		1997									
CN	119600	4		Α		1998	1014	CN	1996-	196954		1	99609	912	
CN	108422	6		В		2002	0508								
EP	874686			A1		1998	1104	EP	1996-	931116		1	99609	912	
EP	874686			B1		2002	0508								
EP	874686			B2		2005	0914								
	R: B	E, DE,	FR,	GB,	IT	, NL									
JP	105116	97		T2		1998	1110	JP	1997-	511710		1	99609	912	
JP	316240	5		B2		2001	0425								
BR	961016	1		Α		1999	0105	BR	1996-	10161		1	99609	912	
RU	218937	_				2002	0920	RU	1998-	107341		1	99609	912	
US	600514	5		Α		1999	1221	US	1998-	43375		1	99808	303	
PRIORITY	APPLN	. INFO).:					FR	1995-	11064		A 1	99509	915	
									1996-	FR1406		W 1	99609	912	
OTHER CO	א וסכים / כי	١.		N/ 7 TO 1	- N - C-1	100	20400	3.0							

OTHER SOURCE(S): MARPAT 126:294889

AB The compds. contain ≥1 divalent metal, at least partially in a reduced state, textured by a phase comprising one or more dopant metals selected from chromium, molybdenum, iron, manganese, titanium, vanadium, gallium, indium, bismuth, yttrium, cerium, lanthanum and other trivalent

lanthanides, in oxide form. When used as catalysts, the metal compds. have an efficiency equivalent to that of Raney cobalt or nickel. They can be used more particularly as hydrogenating catalysts, for hydrogenating various families of nitrogen compds., preferably nitriles.

ANSWER 22 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1997:761967 CAPLUS

DOCUMENT NUMBER:

127:352389

TITLE:

Process for the electrochemical reduction of

organic compounds

INVENTOR(S):

Huber, Gunther; Weiper-Idelmann, Andreas; Kramer,

Andreas; Golombek, Rolf; Frede, Markus; Spiske, Luise;

Schehlmann, Karl Heinz; Steuer, Volker

PATENT ASSIGNEE(S):

SOURCE:

BASF A.-G., Germany Eur. Pat. Appl., 17 pp.

CODEN: EPXXDW

DOCUMENT TYPE:

Patent

LANGUAGE:

German

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PA'	TENT N	O.			KINI)	DATE			API	PLICAT	'ION	NO.		I	DATE
						-									-	
EP	80892	0			A1		1997	1126		ΕP	1997-	1082	24		-	19970521
EP	80892	0			B1		2000	0426								
	R:	ΒE,	CH,	DE,	ES,	FR	GB,	IT,	LI,	, NI						
DE	19620	861			A1		1997	1127		DE	1996-	1962	0861		-	19960523
US	59193	49			Α		1999	0706		US	1997-	8590	34		2	19970520
ES	21464	38			Т3		2000	0801		ES	1997-	1082	24		1	19970521
JP	10046	381			A2		1998	0217		JР	1997-	1336	88		1	19970523
CA	23027	69			AA		1999	0318		CA	1997-	2302	769		1	19970905
WO	99131	32			A1		1999	0318		WO	1997-	EP48	32		1	19970905
	W:	BR,	CA,	CN,	ID,	KR,	MX,	RU,	SG							
PRIORIT	Y APPL	N. I	NFO.	. :						DE	1996-	1962	0861	Α	1	19960523
										WO	1997-	EP48	32	Α	1	19970905

AB The electroredn. of organic compds. is brought about by contacting the organic compound with a cathode consisting of a substrate of elec. conducting porous material (e.g. Raney Ni or Pd on C) and an elec.-conducting, cathodically polarized coating formed on it by in-situ deposition. The cathodically polarized coating consists of a metal, a conducting metal oxide or a carbonaceous material (such as active C), or a mixture of two or more of these materials. The organic compound to be reduced exhibits at least 1 of the following reducible groups or bonds: C-C double bonds, C-C triple bonds, aromatic C-C links, carbonyl groups, thiocarbonyl groups, carboxyl groups, ester groups, C-N triple bonds, C-N double bonds, aromatic C-N links, nitro groups, nitroso groups, and C-halogen simple bonds. Thus, the organic compds. to be electrochem. reduced can be nitriles, dinitriles or dinitro compds.; saturated and unsatd. ketones; and aminocarboxylic acids. electrochem. reduction makes possible, on the one hand, high space-time yields and, on the other hand, a high selectivity in the case of multiply reducible compds. which avoids the formation of hydrogen and can be used on an industrial scale. In an example, a divided electrolytic cell was used with anode and cathode surfaces of 100 cm2 and a filter plate covered with a membrane of steel DIN 1.4571 serving as the cathode. The anode was made of Ti coated with a mixture of Ta and Ir oxides for oxygen evolution. The separation medium was a Nafion-324 membrane. The reaction was carried out discontinuously. A 5% aqueous H2SO4 solution was used as the anolyte. A catholyte was produced, in which vinclozolin

free

[(RS)-3-(3,5-dichlorophenyl)-5-methyl-5-vinyl-oxazoline-2,4-dione] was dissolved in a mixture containing H2O, MeOH, iso-BuOH, and HOAc. Graphite powder was added to the circulating catholyte in a closed cycle and dispersed in the circulating liquid. The deposition was accomplished while the catholyte circuit was closed and the filter outlet was open. The pressure in the cathode chamber rose to 4 + 105 Pa, and the filtrate output amounted to 12 L/h. In the same manner, the catalyst (Degussa type E101N/D, 10% Pd on C) was addnl. deposited. Finally, for 30 min, a d.c. of 20 A was imposed, which required a cell voltage of 35 V at the beginning to 7.5 V at the end of the experiment According to titrimetric determination, 850 ppm Cl- were detected in the discharge (the catholyte was

from Cl- at the start of the experiment), which corresponded to a conversion of 90%. A gas-chromatog. evaluation of the obtained product was made

L8 ANSWER 23 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1996:319149 CAPLUS

DOCUMENT NUMBER: 125:89588

TITLE: Preparation of aliphatic

 α, ω -aminonitriles

INVENTOR(S): Schnurr, Werner; Fischer, Rolf; Bassler, Peter;

Harder, Wolfgang BASF A.-G., Germany

SOURCE: U.S., 3 pp.
CODEN: USXXAM

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT ASSIGNEE(S):

PATENT NO.	KIND DATE	APPLICATION NO.	DATE
		US 1995-370606 DE 1994-4446894	
		WO 1995-EP4984	
RU, SG, SE	C, UA	FI, HU, JP, KR, KZ, MX,	
RW: AT, BE, CH	I, DE, DK, ES, FR,	GB, GR, IE, IT, LU, MC,	NL, PT, SE
AU 9643046	A1 19960719	AU 1996-43046	19951216
EP 800507	A1 19971015	EP 1995-941717	19951216
EP 800507	B1 19990623		
R: AT, BE, CH	I, DE, DK, ES, FR,	GB, GR, IT, LI, LU, NL,	SE, MC, PT, IE
CN 1171777	A 19980128	CN 1995-197138	19951216
CN 1089751			
BR 9510372	A 19980602	BR 1995-10372	19951216
JP 10511371	T2 19981104	JP 1995-520174	19951216
AT 181548	E 19990715	AT 1995-941717	19951216
ES 2135108	T3 19991016	ES 1995-941717	19951216
RU 2153489	C2 20000727	RU 1997-112896	19951216
PL 181529			
CZ 288850		CZ 1997-1977	
BG 63300		BG 1997-101632	
FI 9702761	A 19970626	FI 1997-2761	
PRIORITY APPLN. INFO.:		DE 1994-4446894	
		WO 1995-EP4984	W 19951216

OTHER SOURCE(S): MARPAT 125:89588

AB Aliphatic α, ω -aminonitriles useful for **preparation** of cyclic lactams are **prepared** by partial hydrogenation of aliphatic

 α, ω -dinitriles at elevated temps. and superatmospheric pressure in the presence of a base and of a hydrogenation catalyst, by carrying out the hydrogenation in the presence of ammonia and LiOH or of a compound which gives LiOH during the hydrogenation. Thus, hydrogenation of adiponitrile using Raney nickel catalyst in the presence of ammonia and LiOH for 180 min gave 6-aminocapronitrile with 79.4% selectivity and 93% conversion.

L8 ANSWER 24 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1996:323143 CAPLUS

DOCUMENT NUMBER: 124:342640

TITLE: Process and Group IVB element-doped

Raney nickel catalysts for the

hydrogenation of nitriles into amines

INVENTOR(S): Cordier, Georges; Fouilloux, Pierre; Laurain,

Nathalie; Spindler, Jean Francis

PATENT ASSIGNEE(S): Rhone-Poulenc Chimie SA, Fr.

SOURCE: Fr. Demande, 14 pp.

CODEN: FRXXBL DOCUMENT TYPE: Patent

LANGUAGE: Patent French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 2722784	A1	19960126	FR 1994-9256	19940721
FR 2722784	В3	19960906		

PRIORITY APPLN. INFO.: FR 1994-9256 19940721

OTHER SOURCE(S): CASREACT 124:342640; MARPAT 124:342640 AB Nitriles (e.g., adiponitrile) are hydrogenated into amines

(e.g., hexamethylenediamine) in the presence of a base (e.g.,

NaOH, KOH) in a solvent (e.g., EtOH) using a Raney

nickel catalyst doped with a Group IVB element (e.g., Ti), where

the dopant/Ni weight ratio is 0.05-10%.

L8 ANSWER 25 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1998:377343 CAPLUS

DOCUMENT NUMBER: 129:153993

TITLE: Heterogeneous triple bond electroreduction of

saturated and unsaturated nitriles

AUTHOR(S): Jitaru, Maria; Lessard, Jean; Lowy, Daniel A. CORPORATE SOURCE: Chemistry Research Group, Babes-Bolyai University,

Cluj-Napoca, RO-3400, Rom.

SOURCE: Studia Universitatis Babes-Bolyai, Chemia (1996),

41(2), 70-78

CODEN: SUBCAB; ISSN: 1224-7154 Studia Universitatis Babes-Bolyai

DOCUMENT TYPE: Journal LANGUAGE: English

PUBLISHER:

AB An account on the electroredn. of acrylonitrile to allylamine (AA), and of the catalytic electrohydrogenation of adiponitrile to

hexamethylenediamine (HMDA) is made. In both

synthesis porous nickel Raney electrodes were used as the cathode.

The preparation procedure of the electrodes involved the

co-deposition and electrochem. co-deposition of Ni/Zn and Ni/Al alloys,

followed by the chemical activation of the electrode surface, in alkaline media are described. An improved adherence and compactness of the metallic deposits were achieved by treating the electrode surface with an aqueous

surfactant solution The effects of c.d., supporting electrolyte composition, pH

and temperature on product selectivity were studied. When the **synthesis** of AA was performed in neutral supporting electrolyte, at 288-293 K and at current densities not exceeding 70 mA cm-2, current yields up to 95% were reached. Also, HMDA was obtained with good selectivities ($\geq 85\%$) in a filter press type cell, in neutral supporting electrolytes, at 275-283 K.

REFERENCE COUNT:

18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 26 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1995:789442 CAPLUS

DOCUMENT NUMBER:

123:170544

TITLE:

Method for the catalytic hydrogenation of nitriles

into amines in the presence of doped Raney

nickel catalysts

INVENTOR(S):

Cordier, Georges; Fouilloux, Pierre; Laurain,

Nathalie; Spindler, Jean-Francis

PATENT ASSIGNEE(S):

Rhone-Poulenc Chimie SA, Fr.

SOURCE:

PCT Int. Appl., 18 pp.

CODEN: PIXXD2

DOCUMENT TYPE:

Patent French

LANGUAGE:

rren

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PAT	PATENT NO.			KIND DATE		APPLICATION NO.		DATE	
WO	951809	0		A1	19950706	WO 1994-FR1478		19941216	
	W: B	R, CN,	JP,	US					
	RW: A'	Г, ВЕ,	CH,	DE,	DK, ES, FR,	GB, GR, IE, IT, LU, M	IC, I	NL, PT, SE	
EP	737181			A1	19961016	EP 1995-903847		19941216	
EP	737181			В1	19990224				
	R: B	E, DE,	ES,	FR,	GB, IE, IT,	NL, PT			
CN	114103	1		Α	19970122	CN 1994-194695		19941216	
CN	107580	6		В	20011205				
BR	940846	0		Α	19970805	BR 1994-8460		19941216	
JP	334043	9		B2	20021105	JP 1995-517802		19941216	
US	577716	6		Α	19980707	US 1996-663097		19961125	
PRIORITY	APPLN	. INFO	. :			FR 1993-16008	Α	19931228	
						WO 1994-FR1478	W	19941216	

OTHER SOURCE(S):

MARPAT 123:170544

AB The reduction of nitriles (e.g., adiponitrile) into amines (e.g., hexamethylenediamine) using Raney catalysts doped by ≥1

Group IVb metal (using a doping element/Ni ratio of 0.05-10%)is accomplished by conducting the hydrogenation in a solvent suitable for the nitrile substrate to be hydrogenated, and using at ≥1 alkaline or alkaline-earth metal hydroxide.

L8 ANSWER 27 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1995:905396 CAPLUS

DOCUMENT NUMBER:

123:290375

TITLE:

Preparation of a catalyst for the

hydrogenation of nitriles into amines

INVENTOR(S):

Besson, Michele; Cordier, Georges; Fouilloux, Pierre;

Masson, Jacqueline

PATENT ASSIGNEE(S):

Rhone-Poulenc Chimie SA, Fr.

SOURCE:

PCT Int. Appl., 19 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9517960	A1	19950706	WO 1994-FR1477	19941216
W: BR, CI	I, JP, KR, RU	J, US		
RW: AT, BI	E, CH, DE, DE	<pre> <, ES, FR, </pre>	GB, GR, IE, IT, LU,	MC, NL, PT, SE
EP 737101	A1	19961016	EP 1995-903846	19941216
EP 737101	B1	19980923		
R: BE, DI	E, ES, FR, GE	B, IT, NL		
CN 1139392	Α	19970101	CN 1994-194694	19941216
CN 1082389	В	20020410		
JP 09503439	Т2	19970408	JP 1994-517801	19941216
JP 2851439	B2	19990127		
BR 9408459	A	19970805	BR 1994-8459	19941216
RU 2126297	C1	19990220	RU 1996-116852	19941216
US 5801286	A	19980901	US 1996-663098	19960923
PRIORITY APPLN. IN	O.:		FR 1993-16007	A 19931228
			WO 1994-FR1477	W 19941216

AB Active and selective catalysts of the Raney Ni type for the catalytic hydrogenation of nitriles into amines and, especially dinitriles such as adiponitrile (ADN) into diamines such as diamine hexamethylene (DHM), are prepared by doping an acidic suspension of Raney Ni with a solution of ≥1 metal addition element from transition metal groups, i.e., Groups IVB, VB, and VIB.

L8 ANSWER 28 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1995:909469 CAPLUS

DOCUMENT NUMBER: 123:290374

TITLE: Catalyst preparation for hydrogenating

nitriles into amines

INVENTOR(S): Cordier, Georges; Fouilloux, Pierre; Laurain, Nathalie

PATENT ASSIGNEE(S): Rhone-Poulenc Chimie SA, Fr.

SOURCE: PCT Int. Appl., 25 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. KIND					D DATE	APPLICATION NO.	DATE
WO	9517959			A1	19950706	WO 1994-FR1476	19941216
	W: BR,	CN,	JP,	KR,	RU, US		
	RW: AT,	BE,	CH,	DE,	DK, ES, FR,	GB, GR, IE, IT, LU, MC,	NL, PT, SE
EP	737100			A1	19961016	EP 1995-903845	19941216
EΡ	737100			B1	19980923		
	R: BE,	DE,	ES,	FR,	GB, IT, NL		
CN	1139391			Α	19970101	CN 1994-194680	19941216
CN	1085560			В	20020529		
JP	09505770			T2	19970610	JP 1994-517800	19941216
JP	2851438			B2	19990127		
BR	9408458			Α	19970805	BR 1994-8458	19941216
RU	2131297			C1	19990610	RU 1996-116987	19941216
US	5840989			Α	19981124	US 1996-663099	19960924

PRIORITY APPLN. INFO.: FR 1993-16006 A 19931228

WO 1994-FR1476 W 19941216

AB Active, selective, and stable Raney Ni catalysts for hydrogenation of nitriles into amines are doped with ≥1 transition metal elements, i.e., from Groups IIB, IVB to VIIB of the periodic table, in chelated form. Thus, a precursor Al Ni alloy containing Al at ≤6, preferably <5, especially 2.5-2.4 weight% (Ni basis), is contacted with 6N soda, and also

with
an acidic dopant solution, e.g., of Ti tartrate or Cr tartrate, and the suspension is refluxed in 6N soda for 4 h with intermediate washing with boiling 1-6N soda to obtain the doped catalyst.

L8 ANSWER 29 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1995:534176 CAPLUS

DOCUMENT NUMBER: 123:55064

TITLE: Influence of the medium on hydrogenation of 2-methylglutaronitrile. Selective access to

2-methylpentane diamine or β -picoline after

dehydrogenation

AUTHOR(S): Cordier, Georges

CORPORATE SOURCE: UMR 45 CNRS/RP, Rhone-Poulenc Industrialisation,

Decines, Fr.

SOURCE: Chemical Industries (Dekker) (1995), 62(Catalysis of

Organic Reactions), 285-94

CODEN: CHEIDI; ISSN: 0737-8025

PUBLISHER: Dekker
DOCUMENT TYPE: Journal
LANGUAGE: English

AB A conference. The 2-methylglutaronitrile (I) is obtained as a byproduct of the important adiponitrile production. It, nevertheless, gives rise to interesting chemical transformations. In particular, its hydrogenation can produce 2-methylpentanediamine, a substitute for hexamethylenediamine in polyamide or polyurethane compds. The 3-methylpiperidine, also produced by hydrogenation of I, can be an interesting intermediate for β -picoline production involved in the synthesis of PP vitamin. Using Raney nickel as the catalyst, hydrogenation reactions were performed in various liquid phase compns. 2-Methylpentanediamine was obtained very selectively. The reaction product can be used itself as solvent. Addition of dry ammonia in ethanol in the place of isopropanol / KOH or NaOH medium leads to a mixture of 2-methylpentanediamine, 3-methylpiperidine and some heavy byproducts.

This mixture can be cyclized and dehydrogenated to β -picoline (3-methylpyridine) on a special and very efficient Pd/SiO2 catalyst. The two **processes** have been patented by Rhone-Poulenc.

L8 ANSWER 30 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1993:603018 CAPLUS

DOCUMENT NUMBER: 119:203018

TITLE: Preparation of 6-aminocapronitrile

INVENTOR(S): Sanchez, Kathryn Mary

PATENT ASSIGNEE(S): du Pont de Nemours, E. I., and Co., USA

SOURCE: PCT Int. Appl., 7 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE

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                              19930819
                                         WO 1993-US603
    WO 9316034
                        A1
                                                                19930129
        W: BR, KR
        RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE
    US 5296628 A 19940322 US 1992-836782 19920213
                       A1
    EP 642493
                              19950315
                                         EP 1993-904590
                                                               19930129
    EP 642493
                       B1 19961030
        R: FR, GB
                                         BR 1993-5918
    BR 9305918
                       A 19970916
                                         BR 1993-5918 19930129
US 1992-836782 A 19920213
WO 1993-US603 W 19930129
                                                              19930129
PRIORITY APPLN. INFO.:
                       CASREACT 119:203018
OTHER SOURCE(S):
    Title compound (I) is prepared in high yield and selectivity by
AΒ
    hydrogenation at 200-1000 psig of adiponitrile (II) at
    50-90° in presence of a base, using Raney Ni catalyst and a low
    valent transition metal complex. MeOH, II, aqueous NaOH, Raney Ni and W(CO)6
    were reacted at 500 psi H at 65° to give I. At a conversion of 96%
    selectivity was 88%, whereas at a conversion of <60%, the selectivity to I
    was 100%.
   ANSWER 31 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN
L8
ACCESSION NUMBER: 1993:603015 CAPLUS
DOCUMENT NUMBER:
                       119:203015
TITLE:
                       Process for the preparation of an
                       aminonitrile by partial hydrogenation of a nitrile
                       compound with two or more nitrile groups
                       Bosman, Hubertus Johannes Mechtilda; Vandenbooren,
INVENTOR(S):
                       Franciscus Henricus Antonius Maria Joseph
PATENT ASSIGNEE(S):
                       DSM N. V., Neth.
                       PCT Int. Appl., 16 pp.
SOURCE:
                       CODEN: PIXXD2
DOCUMENT TYPE:
                       Patent
LANGUAGE:
                       English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
                     KIND DATE
                                       APPLICATION NO.
     PATENT NO.
                                                              DATE
                      A1 19930624
                              -----
                                         -----
                                        WO 1992-NL230
    WO 9312073
                                                               19921217
        W: AU, BB, BG, BR, CA, CS, FI, HU, JP, KP, KR, LK, MG, MN, MW, NO, NZ, PL, RO, RU, SD, UA, US
        RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE,
            BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, SN, TD, TG
                       A 19930716 NL 1991-2112
    NL 9102112
                                                               19911218
                             19930719 AU 1993-32689
                                                               19921217
    AU 9332689
                       A1
                                       EP 1993-901480
                                                                19921217
    EP 618895
                       A1
                             19941012
     EP 618895
                       B1
                             19970903
        R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, SE
    JP 07502040 T2 19950302 JP 1992-510801
                                                                19921217
                       E
    AT 157649
                             19970915 AT 1993-901480
                                                                19921217
    ES 2108256
                       T3 19971216
                                         ES 1993-901480
                                                               19921217
    LS 2108256
US 5574181
                                         US 1994-256061 19940817

NL 1991-2112 A 19911218

WO 1992-NL230 A 19921217
                       A 19961112
                                       US 1994-256061
PRIORITY APPLN. INFO.:
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OTHER SOURCE(S): CASREACT 119:203015

AB Aminonitriles were **prepared** by partial hydrogenation of di- or polynitriles in the presence of an alkanolate-treated Group 8 metal-containing catalyst under almost anhydrous conditions. Thus, Degussa BLM 112W Raney Ni was washed with anhydrous MeOH and then stirred with KOMe in MeOH. The

treated catalyst was washed with diaminoethane and then used for hydrogenation of succinonitrile in diaminoethane at 70 atm and 80° while stirring at 1500 rpm for 300 S to give 100% conversion of succinonitrile to a product mixture comprising aminobutyronitrile 85, diaminolactone 14, and pyrrolidine 1 mol %.

ANSWER 32 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1994:30458 CAPLUS
DOCUMENT NUMBER: 120:30458
TITLE: Transfer hydrogenation of nitriles using amine donors

Weigert, Frank J. INVENTOR (S):

PATENT ASSIGNEE(S): du Pont de Nemours, E. I., and Co., USA SOURCE: U.S., 5 pp.

CODEN: USXXAM

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE PATENT NO. US 5237088 A -----A 19930817 US 1992-857344 US 1992-857344 19920325 PRIORITY APPLN. INFO.: US 1992-857344
OTHER SOURCE(S): CASREACT 120:30458; MARPAT 120:30458

R1CN were transfer hydrogenated using R2CH2NH2 [R1, R2= alkyl, X(CH2)y, (CH2) kNMe2, (CH2) mPh, (CH2) nNH(CH2) n+1NH2, (CH2) pNH(CH2) pCN; x = cyano, H2NCH2; k = 2-17; m = 1-17; n, p = 3-11; yr = 3-16] at 20-200° in the presence of Raney Ni and in the absence of H. Thus, hexanenitrile (I) 3.2 g and octylamine (II) 3.1 g were heated at 100° with 3.4 g Raney Ni for 45 min to give a mixture containing I 31, II 48, hexylamine 8.1, and octylnitrile 2.7 area %.

ANSWER 33 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1992:427392 CAPLUS

DOCUMENT NUMBER: 117:27392

Synthesis of non-cyclic aliphatic polyamines
Lin, You Jyh; Schmidt, Stephen R.; Abhari, Ramin TITLE: INVENTOR(S):

W. R. Grace & Co., USA PATENT ASSIGNEE(S):

U.S., 5 pp. SOURCE: CODEN: USXXAM

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

KIND DATE APPLICATION NO. DATE PATENT NO. --------- ------A 19920414 US 1991-709944 19910604 US 5105015 US 1991-709944 PRIORITY APPLN. INFO.:

Polyamines are prepared by hydrogenation of polynitriles in fixed-bed reactors containing Cr- and Ni-promoted Raney Co catalysts in the presence of NH3. Hexamethylenediamine was prepared from adiponitrile in 95.8% selectivity using the above process

ANSWER 34 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1991:582392 CAPLUS

DOCUMENT NUMBER: 115:182392

Intermediates in the catalytic hydrogenation of TITLE:

nitriles

Marion, P.; Grenouiilet, P.; Jenck, J.; Joucla, M. AUTHOR (S): Rhone Poulenc Ind., Decines-Charpieu, 69151, Fr. CORPORATE SOURCE: Studies in Surface Science and Catalysis (1991), SOURCE:

59 (Heterog. Catal. Fine Chem. 2), 329-34

CODEN: SSCTDM; ISSN: 0167-2991

DOCUMENT TYPE: Journal LANGUAGE: English

In the course of the catalytic hydrogenation of α, ω -dinitriles AB

over Raney nickel, byproducts are obtained from C-N

and C-C bond formation. The mechanism of the formation of these compds. was investigated. Cyclic and linear secondary amines can result from the same secondary imine through a transimination process involving a ring-chain tautomerism. Stereochem. results for 2-(aminomethyl)cyclopentylamine (I) are in accord with a specific cyclization pathway favored by an intramol. H bond giving rise to the cis

isomer from aminocapronitrile, unfavored in the case of adiponitrile, which leads to trans-I as the major isomer.

ANSWER 35 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1991:71087 CAPLUS

DOCUMENT NUMBER: 114:71087

The electrochemical synthesis of TITLE:

aminonitriles. I. H-cell studies with

adiponitrile and azelanitrile

AUTHOR(S): Song, Y.; Pintauro, P. N.

Dep. Chem. Eng., Tulane Univ., New Orleans, LA, 70118, CORPORATE SOURCE:

USA

Journal of Applied Electrochemistry (1991), 21(1), SOURCE:

21 - 7

CODEN: JAELBJ; ISSN: 0021-891X

DOCUMENT TYPE: Journal LANGUAGE: English

AR Adiponitrile and azelanitrile were electrochem. hydrogenated to their corresponding aminonitriles in a divided H-cell using Raney nickel powder as the cathode material. The effects of current,

temperature, and solvent/supporting electrolyte composition on product selectivities

were investigated. Syntheses of the fully hydrogenated diamine byproduct increased with increasing current and solution temperature When a 0.8 M adiponitrile/alc./water/ammonium acetate electrolyte was hydrogenated at temps. of 35-45°, 6-aminocapronitrile selectivities in the range of 79-97.permill. and current efficiencies of 50-60.permill. were obtained. The optimum applied current was 60 mA for each 2.5 g of catalyst (an apparent c.d. of 4.8 mA cm-2). For the case of azelanitrile, reaction selectivities for the partially hydrogenated 9-aminononanenitrile product ranged from 80-93%.

ANSWER 36 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

1985:167324 CAPLUS ACCESSION NUMBER:

DOCUMENT NUMBER: 102:167324

TITLE: Production and separation of amines

INVENTOR(S): Cutchens, Charles E.; Mathews, Marion J., III; Sowell,

Mark S., III

PATENT ASSIGNEE(S): Monsanto Co. , USA

SOURCE: U.S., 6 pp.

CODEN: USXXAM

DOCUMENT TYPE: Patent LANGUAGE: English FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. ---------A US 1982-369443 US 1982-369443 US 4491673 19850101 19820419 PRIORITY APPLN. INFO.: 19820419 US 1982-369443

Residual Raney Ni catalyst in the reactor discharge stream during the hydrogenation under pressure of a nitrile to an amine is passified by treating the discharge stream with an inorg. base to form a separable 2-phase mixture, where the first phase contains the amine product and the second phase comprises an aqueous solution containing >40% inorg. base. separable mixture is decanted, purged to remove the Raney Ni catalyst and Al compds., flashed to remove water, and partially recycled back into the inorg. base feed. Thus, crude hexamethylenediamine (I)

[124-09-4], prepared from adiponitrile [111-69-3], containing 301 ppm Na, 9 ppm Al, and 11.6% water was treated with a 55% aqueous NaOH (I-aqueous NaOH ratio of 2:1) at 90° to give I containing 211 ppm Na, <1 ppm Al, and 5.6% water. Decreases in water content, Na, and Al compds. occurred at NaOH concns. 40-70%.

ANSWER 37 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1984:139762 CAPLUS

DOCUMENT NUMBER: 100:139762

TITLE: Catalyst separation in production of amines

INVENTOR(S): Cutchens, Charles E.; Lanier, Lynn H.

PATENT ASSIGNEE(S): Monsanto Co. , USA

SOURCE: U.S., 5 pp.

CODEN: USXXAM

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	US 4429159	Α	19840131	US 1983-476741	19830318
	EP 119980	A1	19840926	EP 1984-870040	19840316
	EP 119980	B1	19860604		
	R: DE, FR, GB,	IT			
	JP 59181242	A2	19841015	JP 1984-50832	19840316
	BR 8401227	Α	19841023	BR 1984-1227	19840316
PRIO	RITY APPLN. INFO.:			US 1983-476741 A	19830318

AB Amines, e.g., hexamethylenediamine (I) [124-09-4], are

prepared by hydrogenation of the corresponding nitriles using H (prepared from CH4 and containing CO2) under pressure in the presence of Raney Ni catalyst in a process in which the amine is discharged into a stream which is separated in the presence of a constant carbonate

(preferably 0.2-0.25 weight %, measured as CO2) to give an upper crude amine stream and a lower catalyst slurry stream. The ratio of alkali to H2O in the catalyst wash water is <0.006. Thus, in the preparation of I from adiponitrile [111-69-3], the carbonate concentration was maintained at <0.6% with catalyst carryover .apprx.35 ppm.

ANSWER 38 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1983:72892 CAPLUS

98:72892 DOCUMENT NUMBER:

TITLE: Catalyst passivation in production of amines

INVENTOR (S): Campbell, Charles R.; Cutchens, Charles E.

Monsanto Co. , USA PATENT ASSIGNEE(S):

SOURCE:

U.S., 5 pp. CODEN: USXXAM

DOCUMENT TYPE:

Patent

LANGUAGE:

FAMILY ACC. NUM. COUNT:

English

PATENT INFORMATION:

US 1980-141926 PATENT NO. KIND DATE APPLICATION NO. --------------US 1980-141836 19800421 US 1980-141836 19800421 US 4359585 Α 19821116 PRIORITY APPLN. INFO.: In the hydrogenation of nitriles to amines over Raney Ni, the catalyst is deactivated, suppressing decomposition of the amine, by adding inorg. bases to the reaction mixts. on discharge from reactors. Thus, when 70 q hexamethylenediamine [124-09-4] was heated with 0.5 g Raney Ni and 0.95 g NaOH under N at 50° for 2 h and refluxed 5 h, the loss of diamine was 0.5%, compared with 31% in the absence of NaOH.

ANSWER 39 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1980:100139 CAPLUS

DOCUMENT NUMBER:

92:100139

TITLE:

Cobalt catalyst

INVENTOR(S):

Uehara, Ryoichi; Horii, Takeo; Imai, Takuya; Tomita,

Yoshiaki; Yamano, Koichiro

PATENT ASSIGNEE(S):

Nikko Scientific and Chemical Industries, Ltd., Japan Jpn. Tokkyo Koho, 4 pp.

SOURCE:

CODEN: JAXXAD

DOCUMENT TYPE:

Patent

LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE	
				•		
JP 54037593	B4	19791115	JP 1975-1138		19741230	
JP 51078795	A2	19760708	JP 1975-1138		19741230	
PRIORITY APPLN. INFO.:			JP 1975-1138	Α	19741230	

AB Raney Co alloys are partially activated by using an aqueous solution of alkali metal borohydride to give a Co catalyst containing Co, Co-Al alloy, and Al hydroxide. The catalyst has good catalytic activity and durability. Thus, a Raney Co alloy (50% Co) 1 kg and a NaBH4 solution (100g/5L) 5L were heated at 80° (for 24 h) to give a slurry-like catalyst. The dried catalyst contained Co 33.2, Al31.1, and B 0.73% (the calculated composition of the

catalyst: Co 19.72, Co-Al alloy 26.96, Al(OH)3 53.32%). The catalyst 2g was added to PhCN 40g (in MeOH 80 mL) and hydrogenation was carried out at 100 kg/cm2 and 124-130° to give PhCH2NH2 32.4g (yield 81.0%, purity 99.8%).

ANSWER 40 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1976:592178 CAPLUS

DOCUMENT NUMBER:

85:192178

TITLE

Catalyst suspension foaming inhibition

INVENTOR(S):

Morgan, Jewel C., Jr. Monsanto Co., USA

PATENT ASSIGNEE(S):

U.S., 5 pp.

SOURCE:

CODEN: USXXAM

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

AUTHOR(S):

PATENT NO. KIND DATE APPLICATION NO. DATE

US 3972940 A 19760803 US 1974-534386 19741219
GB 1473808 A 19770518 GB 1975-51797 19751218
PRIORITY APPLN. INFO.: US 1974-534386 A 19741219

AB Foaming of aqueous suspensions of Raney Ni during catalytic hydrogenation of adiponitrile to hexamethylenediamine (I) was inhibited by adding I to the catalyst suspension.

L8 ANSWER 41 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1963:447798 CAPLUS

DOCUMENT NUMBER: 59:47798
ORIGINAL REFERENCE NO.: 59:8576e-f

TITLE: Synthesis of hexamethylenediamine

from 1,1,1,5-tetrachloropentane Saotome, Kazuo; Miyata, Seiji Asahi Chem. Ind. Co., Tokyo

CORPORATE SOURCE: Asahi Chem. Ind. Co., Tokyo

SOURCE: Kogyo Kagaku Zasshi (1963), 66(2), 205-8

CODEN: KGKZA7; ISSN: 0368-5462

DOCUMENT TYPE: Journal LANGUAGE: Unavailable

AB Synthesis of hexamethylenediamine (I) from

1,1,1,5-tetrachloropentane (II), obtained by the telomerization of C2H4 and CCl4, was carried out via Et δ -cyanovalerate (III) or δ -cyanovaleric acid (IV) and **adiponitrile** (V). III was **prepared** in 91% yield from II (CA 58, 6920e). IV was obtained in

68% yield by adding an equimolar amount of aqueous 20% NaOH to 250 g. III in

250

ml. MeOH (0°, 3 hrs.) and keeping the mixture overnight. V was obtained in 91% yield by passing IV with 7 moles of NH3 through a reaction tube at 350° (boron phosphate was the catalyst; space velocity of reactants was 75 hr.-1). I was obtained in >90% yield by the catalytic hydrogenation of 30 g. V in 100 ml. MeOH and 7.5 g. NH3 under initial H pressure of 90 kg./cm.-2 at 60-70° for 1 hr., with 5 g. Raney nickel and Co as a catalyst.

L8 ANSWER 42 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1957:90332 CAPLUS

DOCUMENT NUMBER: 51:90332 ORIGINAL REFERENCE NO.: 51:16278e-g

TITLE: Preparation of hexamethylenediamine

by hydrogenation of adipic dinitrile on a nickel

catalyst under flow conditions

AUTHOR(S): Freidlin, L. Kn.; Balandin, A. A.; Rudneva, K. G.;

Sladkova, T. A.

SOURCE: Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya

(1957) 166-73

CODEN: IASKA6; ISSN: 0002-3353

DOCUMENT TYPE: Journal LANGUAGE: Unavailable OTHER SOURCE(S): CASREACT 51:90332

AB The effect of various factors on the hydrogenation of adipic dinitrile on a skeleton Ni catalyst is determined Under flow conditions, for a sufficiently long catalyst layer, the yield of hexamethylenediamine attains

80% of the theoretical yield. The high activity of the catalyst and a high ratio of the amount of catalyst to the amount of dinitrile makes it possible to carry out the hydrogenation at 80° and 50 atmospheric H. A decrease in temperature to 60° and in pressure to 20 atmospheric leads to a decrease in the yield of the diamine. An increase in temperature and pressure favors side reactions and increases the rate of deactivation of the catalyst. The addition of 0.24% caustic alkali to the dinitrile decreases the yield of diamine and increases the yield of hexamethyleneimine

ANSWER 43 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1955:87892 CAPLUS

DOCUMENT NUMBER: 49:87892

ORIGINAL REFERENCE NO.: 49:16523i,16524a

TITLE:

Hexamethylenediamine and

hexamethylenediammonium adipate E. I. du Pont de Nemours & Co.

PATENT ASSIGNEE(S): DOCUMENT TYPE: Patent

Unavailable LANGUAGE:

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

CR 73333 APPLICATION NO. DATE -----

19550615 GB 731819 GB

Hexamethylenediamine (I), prepared by the hydrogenation AB of adiponitrile in the presence of a Co catalyst, is purified by fractional distillation to remove 1,2-diaminocyclohexane, hexamethyleneimine, and pentamethylenediamine. After

purification, I can be treated in H2O with an equivalent amount of adipic acid to form an aqueous solution of hexamethylenediammonium adipate (II). These aqueous

solns. have good color stability when stored in contact with air. Conversion of II to the polyamide gives a product of improved color and tensile strength and which is easier to dye with acid dyes.

ANSWER 44 OF 44 CAPLUS COPYRIGHT 2006 ACS on STN

1948:27415 CAPLUS ACCESSION NUMBER:

DOCUMENT NUMBER: 42:27415

ORIGINAL REFERENCE NO.: 42:5848h-i,5849a-c

Reduction of nitriles of dibasic acids over TITLE:

Raney nickel

AUTHOR (S): Arbuzov, B. A.; Pozhil'tsova, E. A.

Bull. acad. sci. U.R.S.S., Classe sci. chim. (1946) SOURCE:

65-70

DOCUMENT TYPE: Journal Russian LANGUAGE:

OTHER SOURCE(S): CASREACT 42:27415

The dinitriles of adipic, sebacic, and succinic acids are readily reduced over Raney Ni at 75-80° to the corresponding amino nitriles under 0.5-0.8 atmospheric H; adiponitrile is reduced to (CH2)6(NH2)2 at 100-10° and 80-100 atmospheric H. The catalyst was made from Al3Ni alloy by treatment with boiling 25% NaOH; after decanting, a fresh batch of alkali was used and the mixture heated 1 h. at 90°; this was repeated 2-3 times and the catalyst was washed free of alkali by H2O and absolute EtOH and was used immediately. Adiponitrile (10 g.), 150 g. BuOH, and 5 g. catalyst were treated with H (0.5-0.8 atmospheric) at 75-80° 10 h.; after filtration and removal of the BuOH in vacuo, the residue was diluted with H2O and 0.5 g. unreacted dinitrile was removed.

Treatment of the residue with BzCl resulted in isolation of 93% Bz derivative of the resulting &- aminocapronitrile, m. 92-3° (from 70% alc.); hydrolysis by HCl gave s-aminocaproic acid-HCl, which gave the free acid, m. 200-2°; the same yield was obtained if the catalyst was added in portions during the course of reduction Succinonitrile (8 g.), 150 cc. BuOH, and 4 g. catalyst similarly treated with H 19 h. at 75-80° gave the Bz derivative of γ aminobutyronitrile (no data given, but a general statement indicated a yield of 90%), as an oil, which on hydrolysis by HCl gave γ-aminobutyric acid, m. 181-2°; HCl salt, m. 134-5°; chloroplatinate, m. 218-20°. Sebaconitrile (10 g.), 150 g. BuOH, and 5 g. catalyst were hydrogenated as above for 26 h.; filtration, removal of the BuOH, and treatment of the residue with BzCl gave (yield not given) the Bz derivative of ε -aminocapric acid, m. 103-5°, which on hydrolysis by HCl gave the free acid, m. 185-7°; chloroplatinate, m. 298-302° (decomposition). Adiponitrile (50 g.), 450 g. BuOH, and 17 g. catalyst heated $2\ h.$ to 110° at 90 atmospheric initial H pressure (45 atmospheric final) gave 1.3 g. unreacted dinitrile,

and 46 g. (85.7%) hexamethylenediamine, b. 200-4°; 3.5 g. of tar was formed. Increase of the temperature to 150° (14 h.) dropped the yield to 26.8%; halving of the catalyst amount dropped the yield to 64%. The high-pressure runs were made in a rotating Bergius autoclave.

=> log y		
COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	159.80	160.01
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL
	ENTRY	SESSION
CA SUBSCRIBER PRICE	-33.00	-33.00

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